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## INFORMATION REPORT

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SUPPLEMENT TO  
REPORT NO. 

1. The Karpov Institute is located in a garden plot at No. 10 Obukha Street, in the Molotov district of Moscow (55-45N, 37-35E). A nitrogen research institute adjoins the installation on the south. Residential blocks border the eastern fence of the installation, and adjoining the eastern fence is a building housing a factory producing electrical equipment for medical use. An institute of the Moscow Military Engineer Academy is located northwest of the entrance to the Karpov Institute, on the opposite side of Obukha Street. A streetcar line runs from the Kursk railroad station, located about 800 meters east-north-east of the installation, along Obukha Street, to the center of the city, crossing a bridge over the Moskva River.
2. The designation of the institute is the Khimicheskiy Nauchno-Issledovatel'skiy Institut imeni Karpova (Karpov Chemical Scientific Research Institute). Part of the research work done at the Institute was controlled by the Ministry for the Chemical Industry. Administratively, the Institute is controlled by Chemical Plant No. 94, located near the southern part of Moscow.
3. The area in which the two buildings of the Institute are located is somewhat more than 200 x 150 meters. The older building was constructed in 1928 and the northern building was constructed even earlier. In 1947 or 1948 an annex was built and was equipped with an experimental installation for a pressure of 700 atmospheres, used in the production of heavy water ( $D_2O$ ), which was installed along the western wall of the southern building.
4. After 1 November 1946, a group of German chemists from the Leuna Works were compulsorily assigned to the Karpov Institute. The chemists, most of them with their dependents, were accommodated in a recreation house of the Ministry

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for the Chemical Industry at No. 1 Karl Marx Street, Babushkin (55-52N, 37-42E), a northern suburb of Moscow. They went to the Institute daily by bus.

5. Personalities contacting the German experts concerning employment and personnel problems included the following: in 1946, Kazatkin (fmu); starting in 1947, Novikov (fmu), in his capacity as deputy minister, and Professor Planovskiy (fmu); and in 1948, Shushkin (fmu), the secretary of the deputy minister, and, on personnel matters, Romanov (fmu) and Ofitserov (fmu). The Soviet officials who supervised the German chemists working at the Karpov Institute included the following: Professor Zhavoronkov (fmu), director of the Institute; Professor Shukhevitskiy (fmu), deputy director and head of the special technical office; Professor Medvedev (fmu), an expert on oxidation kinetics and organic and inorganic oxidation, who allegedly was one of the leading experts of the Institute and corresponded with American and British scientists; Professor Kagan (fmu), an expert on reaction kinetics; Professor Petrianov (fmu), an expert on heavy water, who was assigned, as a colonel, to the Leuna Works from 1945 to 1948; Professor Boreskov (fmu), an expert on catalysis; Shutikov (fmu), administrative director and head of Department No. 1, as well as political commissar; and Engineer Kagan (fmu), supervisor of the German group.
6. Before starting their work at the Institute, all the Germans had to sign secrecy agreements. In November 1946 the experts were assigned to the following fields:
  - a. Group Leader - Dr. Ernst Paul Herold, a chemist and the former director of the organic department, which included an experimental laboratory, at the Leuna Works.
  - b. Heavy Water - Dr. Karl Hermann Geib, a physicochemist; Dr. Heinrich Elm, a physicochemist, formerly employed in the heavy water installation of the Leuna Works; Graduate Engineer Dr. Karl Bode, who had worked on the production of heavy water at the Leuna Works; and Professor Petrianov (fmu), Soviet scientific caretaker.
  - c. Hydrogen Peroxide - Dr. Luis Gomassmer, a chemist and expert in processing techniques; Dr. Hans-Joachim Froelich, a chemist and analyst; Dr. Helmut Jochinks, an expert who specialized in organic chemistry and whose former field at the Leuna Works was propane oxidation; and Professor Medvedev (fmu), Soviet scientific caretaker.
  - d. Catalysts - Dr. Kurt Kosterhon, an inorganic chemist; Dr. Walter Schmidt, a chemist; and Professor Boreskov (fmu), Soviet scientific caretaker.
  - e. Plastics - Dr. Wilhelm Falkenberg, a chemist who specialized in the field of plastics.
  - f. Rocket Fuels - Dr. Friedrich Asinger, an organic chemist, who formerly was the head of a test laboratory at the Leuna Works; Dr. Franz Scheuer, a physicist; Dr. Fritz Andreas, an organic chemist; and Professor Kagan (fmu), Soviet scientific caretaker.
7. Practical work at the laboratories first started in January 1947, when the laboratory equipment from the Leuna Works arrived and was installed in the Institute.
8. The German experts could work with heavy water only in the laboratories. In pursuance of the activities started in Bitterfeld, Germany, heavy water was to be obtained through the use of an evaporation installation. Allegedly, no final results were obtained because the team soon split-up. Dr. Elm was assigned to the rocket fuel team and Dr. Geib's activities were confined to the writing of reports on hydrogen isotopes and atomic hydrogen. After the

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German scientists were dismissed, research work on heavy water was continued at the Institute.

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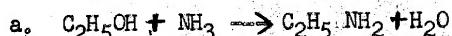
9. Several series of tests were started to obtain pure hydrogen peroxide by the oxidation of propane ( $C_3H_8$ ), but the tests failed because of the lack of necessary equipment. It was impossible to obtain the required purity, namely, 98.5 percent propane. Hence, activities were confined to examining possibilities of obtaining hydrogen peroxide from the oxidation product of propane gas ( $CH_3 \cdot CH_2 \cdot CH_3$ ) as aldehydes were formed, during the oxidation, at a rate of about 25 percent. Simple distillation, even under lowered pressure, resulted in explosions. Most vacuum distillation tests also failed. In addition, attempts were made to combine  $H_2O_2$  with hydrate-forming inorganic salts and to separate the aldehydes again after washing these salts. In this connection, experiments were made using alum ( $K_2Al_2(SO_4)_4 \cdot 24H_2O$ ) and potassium fluoride ( $KF \cdot 2H_2O$ ). These experiments failed, as did the attempts to achieve the desired results using the slightly soluble acetone peroxide (acetone -  $CH_3COCH_3$ ). It was only by the use of calcium peroxide ( $CaO_2 \cdot 8H_2O$ ) that a possibility of developing a method suitable for technical evaluation was discovered. The manufacturing process was as follows: The oxidation product of propane gas was diluted with three parts water by volume. Finely pulverized calcium oxide ( $CaO$ ), passed through a sieve of 40,000 meshes per sq cm, was added to this solution in small quantities, at a temperature of 0° centigrade and with constant stirring, at a rate of about 1.1 mol  $CaO$  per 1 mol  $H_2O_2$ . The slightly soluble calcium peroxide was separated from the solution by a suction filter and was repeatedly washed with moderately cold water to remove the last traces of aldehydes and calcium salts of the organic acids. The moist calcium peroxide was subsequently broken up into  $H_2O_2$  and calcium sulphate ( $CaSO_4$ ), by means of dilated sulphuric acid of about 20 percent. After washing the calcium sulphate paste, a hydrogen peroxide solution of 1.5 to 2 percent was obtained, which was subsequently processed by vacuum distillation. However, a small amount of calcium sulphate would always remain in the solution. The tests were performed with a synthetic mixture of hydrogen peroxide, acetic aldehyde ( $CH_3-CHO$ ), and formaldehyde ( $HCHO$ ). No results were obtained from attempts to separate hydrogen peroxide by electro-osmosis, diaphragm diffusion, and other methods. A series of analyses were made to ascertain the exact fractions of  $H_2O_2$ , acetic aldehyde, and formaldehyde in the oxidation product of propane. The Soviet authorities displayed little interest in these activities, which were terminated, in May 1947, by Soviet order.

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10. In the field of catalysis, tests with hydration catalysts of nickel were performed at the laboratory of Professor Boreskov. The tests allegedly were an attempt to separate heavy and light hydrogen by hydration.

11. The German expert on plastics, who was employed at the Karpov Institute from November 1946 to February 1947, was allowed to do only research work and later was assigned to plastics factories in the USSR.

12. In an attempt to obtain rocket fuels, several series of tests were started at the Karpov Institute using various amines. The chemists tried to manufacture triethylamine,  $(C_2H_5)_3N$ , by a synthesis of ammonia ( $NH_3$ ) and alcohol ( $C_2H_5OH$ ):  $3C_2H_5OH + NH_3 \rightarrow N(C_2H_5)_3 + 3H_2O$ . The process was performed in two steps, starting with monoethylamine ( $C_2H_5NH_2$ ) and diethylamine ( $(C_2H_5)_2NH$ ):

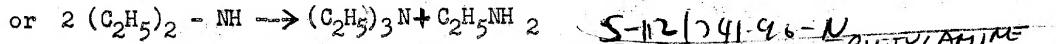
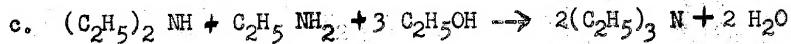


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OF PRODUCING TRIETHYLAMINE FROM AMMONIA AND ETHYL ALCOHOL

The yield of triethylamine obtained in one working process was too little, as concentrated ethylene -  $(CH_2: CH_2)$  was also formed. Moreover, distilling proved to be difficult when applying this method, since triethylamine and alcohol formed a compound which vaporized simultaneously with the desired end product. In the summer of 1948 the method had been improved to such an extent that it was proposed to build an appropriate technical installation at a place on Lake Baikal. Chrome-nickel-ferrous contact materials were used as catalysts. During the designing work on the technical installation at the Karpov Institute, Candidate of Chemistry Gelstein (fmu) was assigned to the German expert as a Soviet assistant. When the German team was transferred, in the summer of 1948, to Dzerzhinsk (56-15 N, 43-24 E), this installation was moved to Chemical Plant No. 94 in Moscow. The Karpov Institute attempted to find cheap raw materials, other than ethyl alcohol and ammonia, suitable to use in producing rocket fuels on the basis of amine. Having succeeded in synthesizing acetonine ( $C_6H_{18}N_2$ ) from acetone ( $CH_3COCH_3$ ) and ammonia ( $NH_3$ ), the German experts suggested the acetone could be obtained from gases obtained by cracking petroleum over propylene ( $CH_2CHCH_3$ ) and isopropyl alcohol ( $(CH_3)_2CHOH$ ). The Soviets, however, insisted that they were unable to produce acetone by such a method and would obtain their requirements of acetone by fermenting Indian corn as they had before. The main hindrance in obtaining acetone was the inability to obtain a clean fraction of propane and propylene by distillation. Although acetone was hard to obtain, the Soviets seemed to be interested in acetonine, which was referred to as product K at the Institute. A technical installation, designed for this purpose at the Institute, was built in Chemical Plant No. 94 in Moscow. Preparatory work had been completed by the spring of 1947. Several tons of acetonine were manufactured, but it was not ascertained whether continuous production was started. The method used at the laboratories of the Karpov Institute to make acetonine is as follows: three liters acetone and, as a catalyst, 30 gr ammonium nitrate ( $NH_4NO_3$ ) were poured in a five-liter retort with a reflux cooler, and enough ammonia introduced, for a period of six to ten hours, to saturate the solution. The temperature rises from 30° to 45° centigrade. After cooling, the contents were shaken once with a 20 percent solution of caustic soda and once with a 40 percent solution of caustic soda; subsequently, the solutions were separated. Potassium hydroxide (KOH) was used as the dehydrating agent. The product obtained, consisted of about three percent mesityl oxide, 90 percent acetonine, and seven percent of the higher amines, and was used for the fuel mixtures. The acetonine was identified, in the summer of 1947, by the German expert entrusted with the analysis, as penta-methyl-tetra-hydro-pyrimidine. The acetonine showed good ignition-delaying capacity. However, its viscosity was too high and when air was admitted crystals formed by hydration, as the liquid had a great capacity to absorb moisture from the air. The formation of hydrates was reduced, but not eliminated, by adding benzines or other amines. Therefore, a large-scale study of the hydration and fractions of acetonine was made and acetonine-like amines and others which were obtainable from acetone and known previously were considered. 2,4-diamino -  $N_4$  -isopropyl-2-methylpentane and 2,4-diamino-2-methyl-pentane proved to be better than acetonine. In contrast with acetonine, the two amines were distillable in an undecomposed condition. However, 2,5-dihydro-, 2,4,6-tetramethylpyridin showed poorer properties than acetonine. Diacetone-amine, diacetone-diamine, and triacetone-amine (triacetine =  $CH_3.CO.O.CH(CH_2.O.CO.CH_3)_2$ ) also proved to be hard to ignite. The directing agencies emphasized that certain materials were not available, and did not lend support to these tests. Nitric acid was used in the process to determine the inflammability of the amines. All mixtures which ignited when mixed with

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nitric acid were called Hypergoles (code name for carbon derivatives). The criterion on the serviceability of the mixtures was based on the length of time required for ignition of the individual materials after coming in contact with nitric acid. This interval was referred to as ignition delay. The fixed limits for serviceability ranged from 5 and  $30 \times 10^{-3}$  seconds. In the testing compartment, products with too short an ignition delay had too high an explosive effect, whereas those with too long an ignition delay burned too slowly. Such a testing compartment allegedly existed at Chemical Plant No. 94 in Moscow, as one or two kg samples of mixtures, whose ignition delays were within the established limits, were forwarded there. In two or three cases, 100 to 200 kg specimens were also demanded by that agency. The measuring was performed by a German expert at the laboratory of the Institute. In the beginning, the measuring was done with the aid of a motion picture camera. This method did not seem to be precise and was expensive. Therefore, an electrical apparatus which could perform 30 to 40 measurements per hour was constructed. Three types of nitric acid were available as oxidizing agents for the ignition-delay measurements: Acid A, a nitric acid of 98 to 99.5 percent; Acid B, a nitric acid of 98 to 99.5 percent with one percent ferric chloride ( $FeCl_3$ ); and Acid C, a nitric acid of 98 to 99.5 percent with ten percent sulphuric acid. Acid B was the most suitable, as the ferric chloride had a catalytic effect. However, Acid B was so strongly corrosive, that it was unsuitable for technical use. Acid C had the lowest corrosive effect, but it was the slowest to react. The time of ignition delay was successfully shortened by adding a ferrous compound to the amine solution. In the beginning, ferrous pentacarbonyl ( $Fe(CO)_5$  = ferrous carbonyl) was added, but it decomposed in the amine solution after several hours. Iron soap was also tried. Ferrous tributyrate proved to be the best ferrous compound. The catalytic properties of other heavy metals such as chromium, nickel, tungsten, vanadium, lead, copper, silver, cobalt, arsenic, molybdenum, and zinc were checked. They were used partly as butyrates and partly as acetylacetonate ( $CH_3COCH_2COCH_3$ ) compound; the application of the latter compound led to the formation of metallic salts. The final conclusion of these investigations was that iron was the best catalyst, and slightly exceeded chromium. All other metals were far inferior or had too delayed a reaction. The ferrous butyrate was made of ferric chloride and sodium butyrate in an alcohol-water-benzole solution. In 1948 tests were performed at the Karpov Institute to determine if sulphur compounds would be used to manufacture rocket fuels. However, the actual tests were performed at the OKA plant in Dzerzhinsk after the German experts had been transferred there.

Attachments: Two

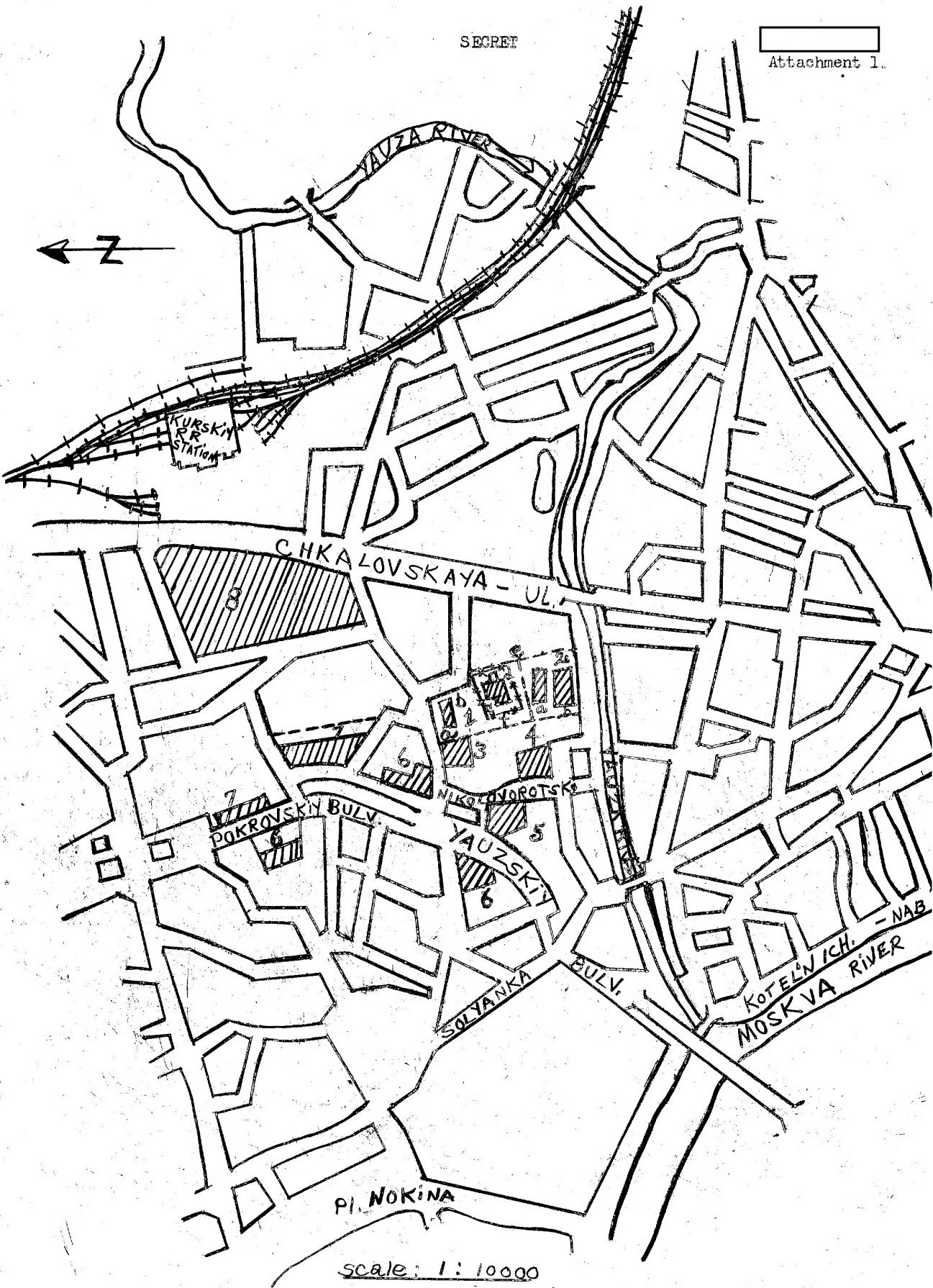
1. Location and layout sketch of the Karpov Institute
2. Sketch of the apparatus for measuring the ignition delay of rocket fuels.

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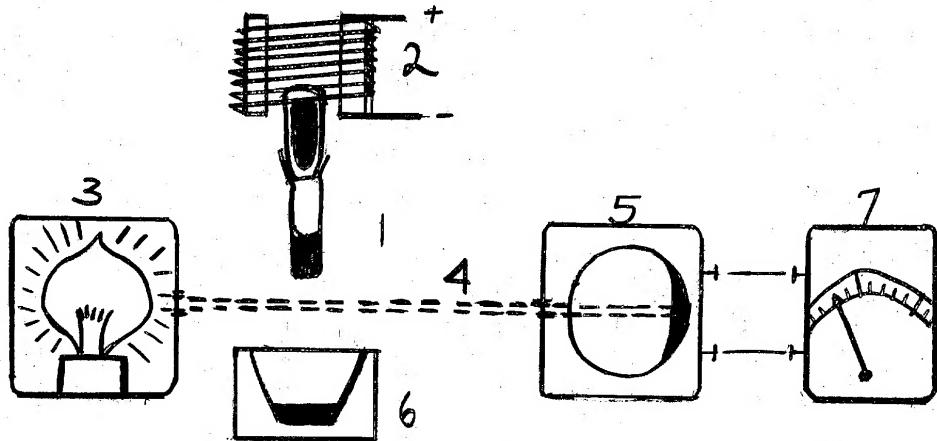
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Attachment 1.



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Attachment 2.

Apparatus Used to Measure the Ignition Delay of Rocket FuelsNot to ScaleLegend:

1. Pipette.
2. Coil.
3. Light
4. Light Ray.
5. Potash or selenium cell.
6. Crucible containing 1 cubic cm of material, used as catalyst.
7. Ballistic galvanometer.

Explanation of the Apparatus Used to Measure the Ignition Delay of Rocket Fuels

About one cubic cm of nitric acid is put into the pipette. The top of the pipette is closed by a glass stopper with a fused iron core. A crucible containing one cubic cm of the material used as catalyst is placed below the pipette. By closing a circuit, the stopper is drawn up into the coil, opening the pipette. The nitric acid drops and cuts the light ray, which registers on the potash or selenium cell. The nitric acid and the amine solution ignite in the crucible, and the ignition again interrupts the light ray, which is again registered by the cell. The cell reactions are indicated on a ballistic galvanometer. The amplitude of the galvanometer makes it possible to measure the amount of time elapsing between the first and the second impulses. The device was calibrated by using a microchronometer with a film. The measuring range is 10 to  $1200 \times 10^{-4}$  seconds and the limit of error is  $\pm 10 \times 10^{-4}$  seconds.

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Attachment 1

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Legend:

1. Karpov Chemical Research Institute.
  - a. Entrance at No. 10 Obukha Street.
  - b. Old building of the Institute, referred to as the upper building ("Upper Korpus").
  - c. So-called lower building ("Lower Korpus") of the Institute, constructed in 1928.
  - d. Annex building constructed in 1947 and/or 1948.
  - e. Board fence.
  - f. Closely guarded fence.
2. Nitrogen research institute, referred to as the Azotnyy Institute.  
The terrain sloped rather steeply to the south, toward the Yauza River.
  - a. New building of the institute.
  - b. Older building, which formerly housed a sugar factory.  
A number of small sheds, not indicated in the sketch, were also located in the area of the two institutes.
3. Factory producing equipment for medical use.
4. Branch of a Moscow automobile factory.
5. Electrotechnical factory.
6. Three institutes of the Military Engineer Academy.
7. Two barracks installations.
8. Prison.

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